

# BENEATH THE SURFACE: X-RAY ANALYSES OF BATTERY MATERIALS AND STRUCTURES

*A Battery Webinar Series by Rigaku*

## Non-destructive Elemental Analysis of Batteries Using XRF

*Starting at 1 pm CDT*



- *You will be muted during the webinar.*
- *You can ask questions using the Q&A tool.*
- *You should hear music if your sound is working.*

# BENEATH THE SURFACE: X-RAY ANALYSES OF BATTERY MATERIALS AND STRUCTURES

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## Non-destructive Elemental Analysis of Batteries Using XRF

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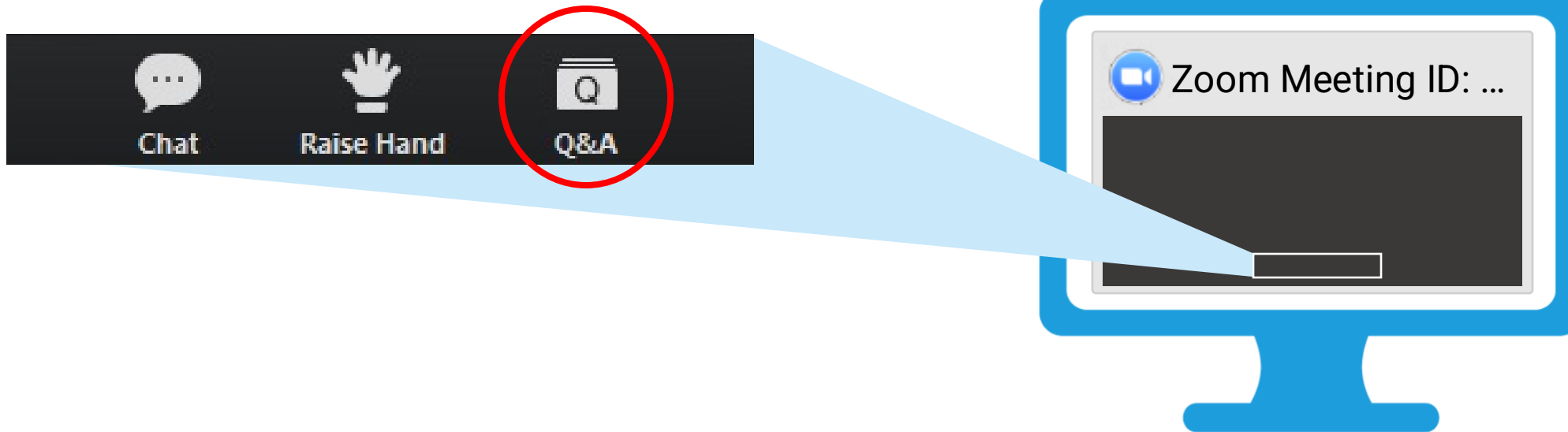


*We are starting now...*

Presenter: **Amber Quevy** | XRF Applications Lab Manager

Co-presenter: **Tim Bradow** | Sr. Business Development Manager

Host: **Aya Takase** | Head of Global Marketing



You can ask questions during the presentation.  
Please use the Q&A to ask questions.



Recording will be available tomorrow.



# Non-Destructive Elemental Analysis of Batteries Using XRF

# You will learn

1. What is XRF?
2. Why use XRF?
3. Where is XRF used?
4. How is XRF used? **Examples!**



# Polling Question #1



Microsoft Stock



# What is XRF?

# The Subject

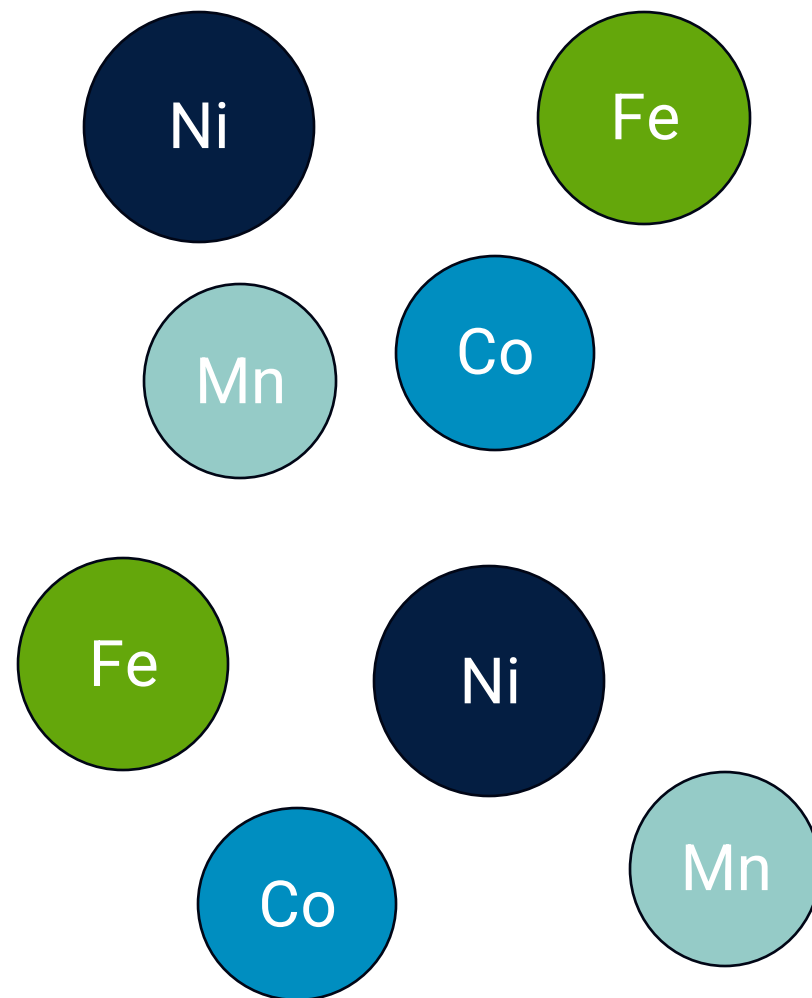


# The Building Blocks

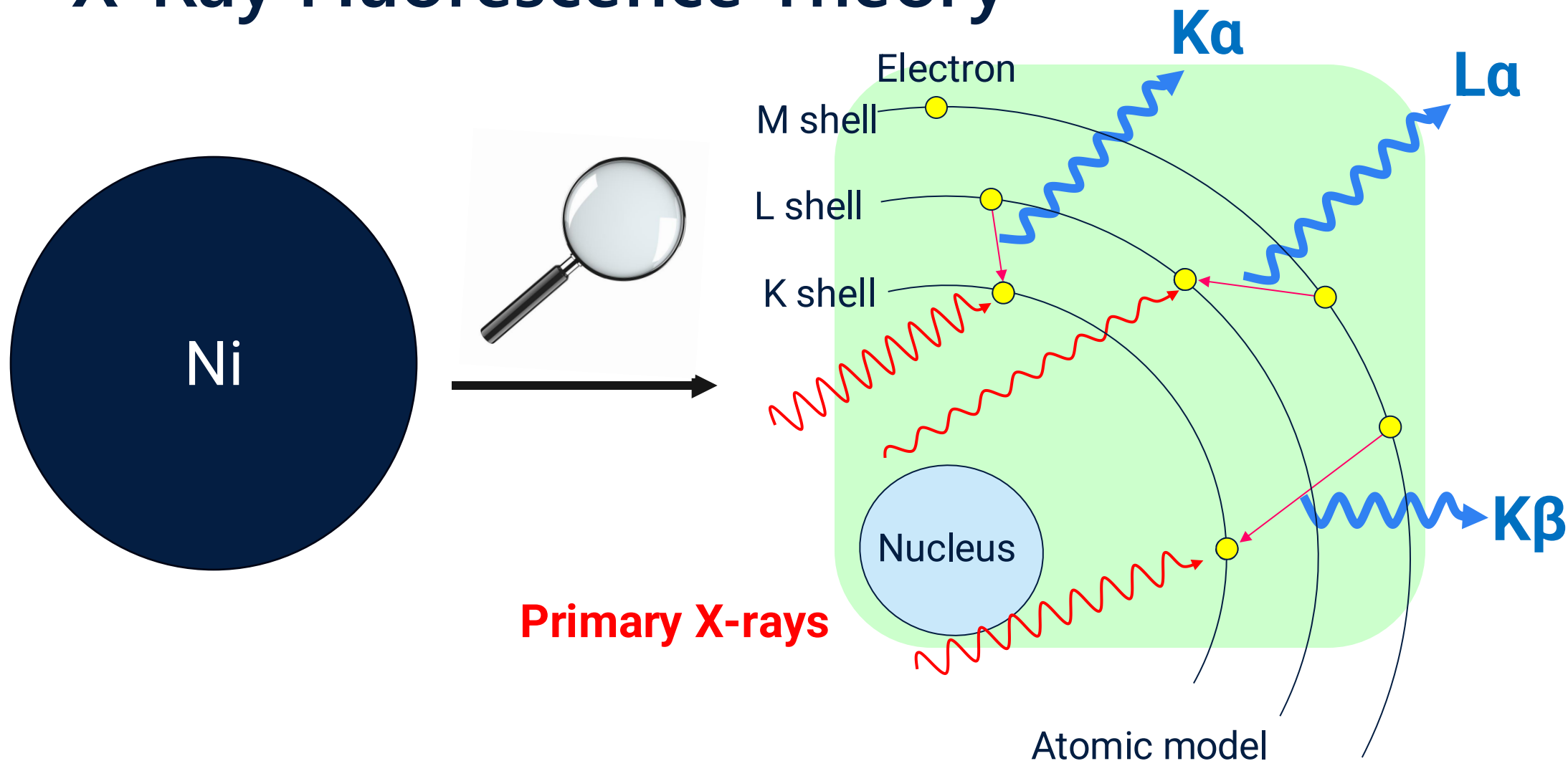
## Periodic Table of Chemical Elements

1 Ia																		18 0																	
1 H 1.008		2 IIa																		2 He 4.0026															
3 Li 6.94		4 Be 9.0122 Ka RX85 0.111																		5 B 10.81 Ka RX85 0.19		6 C 12.011 Ka RX61 0.252		7 N 14.007 Ka RX45 0.4		8 O 15.999 Ka RX35 0.51		9 F 18.998 Ka RX25 0.664		10 Ne 20.180					
11 Na 22.990 Ka RX25 1.07		12 Mg 24.305 Ka RX25 1.30																		13 Al 26.982 Ka PET 1.55		14 Si 28.085 Ka PET 1.83		15 P 30.974 Ka Ge 2.14		16 S 32.06 Ka Ge 2.47		17 Cl 35.45 Ka Ge 2.82		18 Ar 39.948					
19 K 39.098 Ka LIF(200) 3.742 136.69 3.59		20 Ca 40.078(4) Ka LIF(200) 3.359 113.09 4.00		21 Sc 44.956 Ka LIF(200) 0.8302 4.49		22 Ti 47.867 Ka LIF(200) 0.7873 22.54 18.0		23 V 50.942 Ka LIF(200) 0.7476 21.39 19.0		24 Cr 51.996 Ka LIF(200) 0.6296 5.98		25 Mn 54.938 Ka LIF(200) 0.5493 6.54		26 Fe 55.845(2) Ka LIF(200) 0.5445 7.10		27 Co 58.933 Ka LIF(200) 0.5277 7.71		28 Ni 58.693 Ka LIF(200) 0.4865 8.29		29 Cu 63.546(3) Ka LIF(200) 0.4501 8.86		30 Zn 65.38(2) Ka LIF(200) 0.4178 9.65		31 Ga 69.723 Ka LIF(200) 0.3690 10.4		32 Ge 72.630(8) Ka LIF(200) 0.3486 11.1		33 As 74.922 Ka LIF(200) 0.3187 11.9		34 Se 78.971(8) Ka LIF(200) 0.2866 12.7		35 Br 79.904 Ka LIF(200) 0.2653 13.5		36 Kr 83.798(2)	
37 Rb 85.468 Ka LIF(200) 0.9269 26.60 15.7		38 Sr 87.62 Ka LIF(200) 0.8766 25.13 16.1		39 Y 88.906 Ka LIF(200) 0.8302 23.78 17.0		40 Zr 91.224(2) Ka LIF(200) 0.7873 22.54 18.0		41 Nb 92.906 Ka LIF(200) 0.7476 21.39 19.0		42 Mo 95.95 Ka LIF(200) 0.7107 20.32 20.0		43 Tc 99 Ka LIF(200) 0.674 19.33 21.1		44 Ru 101.07(2) Ka LIF(200) 0.6445 18.41 22.1		45 Rh 102.91 Ka LIF(200) 0.6147 17.55 23.2		46 Pd 106.42 Ka LIF(200) 0.5869 16.75 24.4		47 Ag 107.87 Ka LIF(200) 0.5608 16.00 25.5		48 Cd 112.41 Ka LIF(200) 0.5365 15.30 26.7		49 In 114.82 Ka LIF(200) 0.5136 14.64 27.9		50 Sn 118.71 Ka LIF(200) 0.4920 14.03 29.1		51 Sb 121.76 Ka LIF(200) 0.4718 13.45 30.4		52 Te 127.60(3) Ka LIF(200) 0.4528 12.90 31.8		53 I 126.90 Ka LIF(200) 0.4348 12.39 33.2		54 Xe 131.29	
55 Cs 132.91 Ka LIF(200) 0.4018 11.44 35.9		56 Ba 137.33 Ln LIF(200) 2.776 67.13 5.99		57-71 Lanthanoids		72 Hf 178.49(2) Ln LIF(200) 1.569 45.86 11.3		73 Ta 180.95 Ln LIF(200) 1.522 2.463 11.7		74 W 183.84 Ln LIF(200) 1.476 43.00 12.1		75 Re 186.21 Ln LIF(200) 1.433 40.00 12.5		76 Os 190.23(3) Ln LIF(200) 1.391 40.11 13.0		77 Ir 192.22 Ln LIF(200) 1.352 39.20 13.4		78 Pt 195.08 Ln LIF(200) 1.313 38.04 13.9		79 Au 196.97 Ln LIF(200) 1.277 36.94 14.4		80 Hg 200.59 Ln LIF(200) 1.242 35.99 14.8		81 Tl 204.38 Ln LIF(200) 1.207 34.86 15.3		82 Pb 207.2 Ln LIF(200) 1.175 32.99 15.8		83 Bi 208.98 Ln LIF(200) 1.144 32.99 16.4		84 Po 210 Ln LIF(200) 1.114 32.10 16.9		85 At 210 Ln LIF(200) 1.085 31.25 17.5		86 Rn 222	
87 Fr 223 Ln LIF(200) 1.0308 29.64 19.6		88 Ra 226 Ln LIF(200) 1.009 28.88 19.3		89-103 Actinoids		104 Rf 267		105 Db 268		106 Sg 271		107 Bh 272		108 Hs 277		109 Mt 276		110 Ds 281		111 Rg 280		112 Cn 285		113 Nh 278		114 Fl 289		115 Mc 289		116 Lv 293		117 Ts 293		118 Og 294	
57-71 Lanthanoids		57 La 138.91 Ln LIF(200) 2.665 82.98 6.26		58 Ce 140.12 Ln LIF(200) 2.561 79.98 6.54		59 Pr 140.91 Ln LIF(200) 2.463 75.40 6.83		60 Nd 144.24 Ln LIF(200) 2.370 72.10 7.12		61 Pm 145 Ln LIF(200) 2.283 69.03 7.45		62 Sm 150.36(2) Ln LIF(200) 2.199 66.20 7.73		63 Eu 151.96 Ln LIF(200) 2.120 63.54 8.04		64 Gd 157.25(3) Ln LIF(200) 2.046 61.08 8.37		65 Tb 158.93 Ln LIF(200) 1.976 58.77 8.70		66 Dy 162.50 Ln LIF(200) 1.909 56.57 9.03		67 Ho 164.93 Ln LIF(200) 1.845 54.52 9.38		68 Er 167.26 Ln LIF(200) 1.765 52.58 9.73		69 Tm 168.93 Ln LIF(200) 1.689 50.77 10.1		70 Yb 173.05 Ln LIF(200) 1.612 49.04 10.5		71 Lu 174.97 Ln LIF(200) 1.572 47.41 10.9					
89-103 Actinoids		89 Ac 227 Ln LIF(200) 0.980 29.15 19.8		90 Th 232.04 Ln LIF(200) 0.956 27.45 20.5		91 Pa 231.04 Ln LIF(200) 0.933 26.78 21.1		92 U 238.03 Ln LIF(200) 0.911 26.13 21.7		93 Np 237		94 Pu 239		95 Am 243		96 Cm 247		97 Bk 247		98 Cf 252		99 Es 252		100 Fm 257		101 Md 258		102 No 259		103 Lr 262					

# Bob's Close-up



# X-Ray Fluorescence Theory

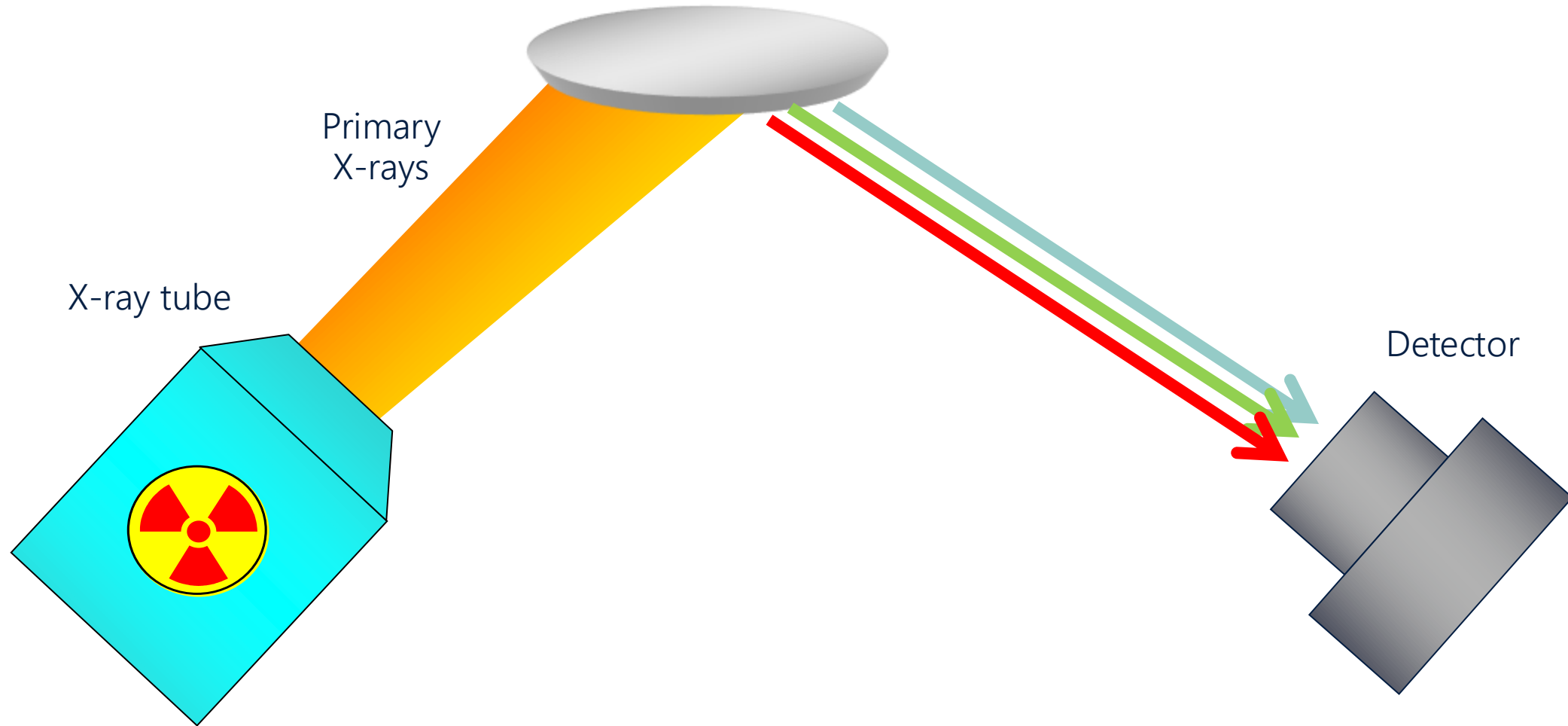


# X-ray Fluorescence Types: EDXRF vs. WDXRF

Instrument	EDXRF	WDXRF
Format	Handheld or Benchtop	Benchtop and Floor models
Tube	Ag, Pd, Rh	Pd, Rh
Wattage	4W-100W	200W-4000W
Analysis range	Na-U	Be-Cm
Atmosphere Options	Air, Helium, Vacuum	Helium, Vacuum
Cost	\$-\$\$	\$\$-\$\$\$



# Types of XRF: Energy Dispersive (EDXRF)



# Examples of EDXRF Instruments



NEX QC  
Series

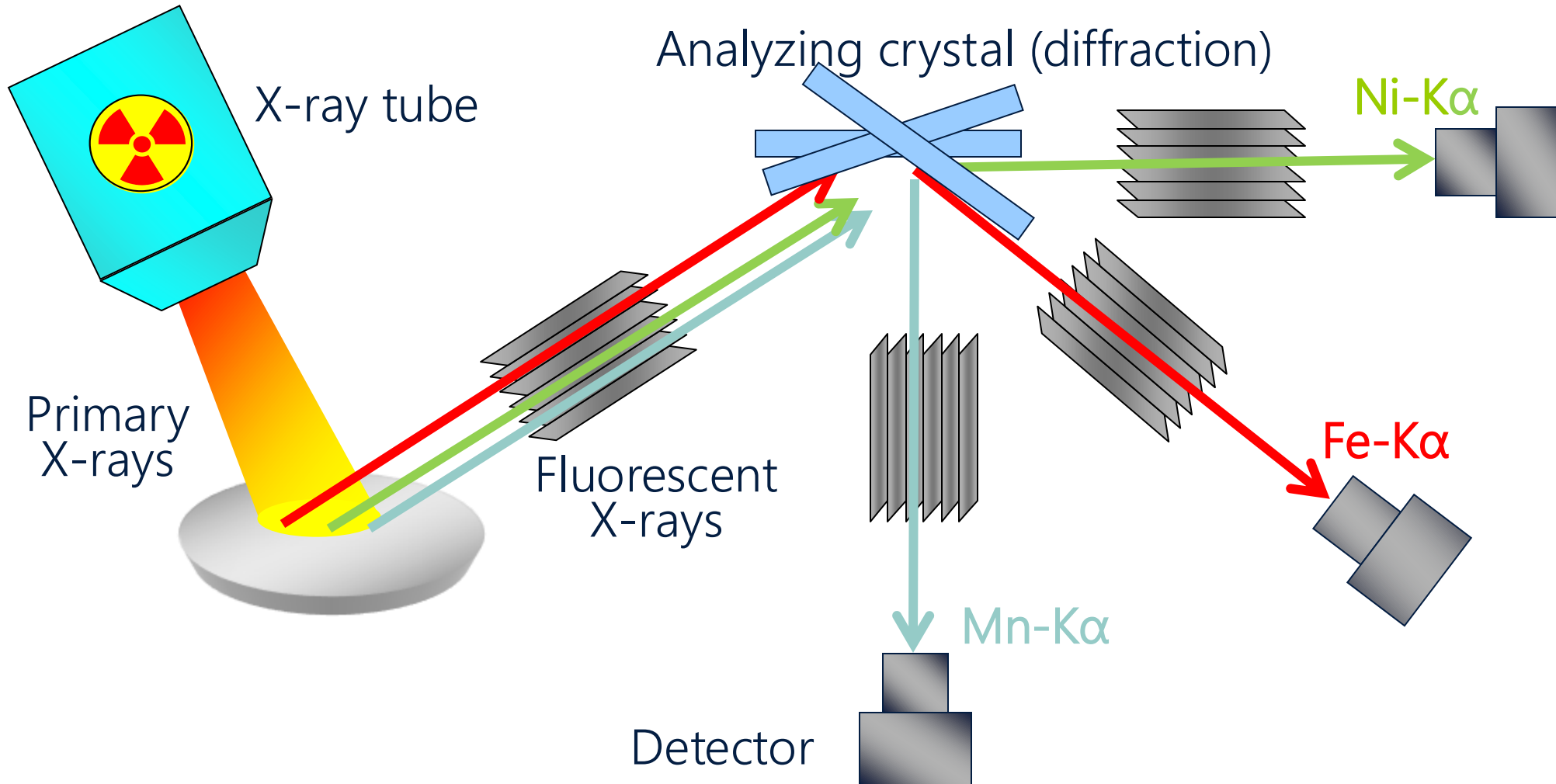


NEX DE  
Series



NEX CGII  
Series

# Types of XRF: Wavelength Dispersive (WDXRF)



# Examples of WDXRF Instruments



Supermini200



ZSX Primus Series

# Polling Question #2



Microsoft Stock

# Why use XRF?



# Traditional Analysis Techniques

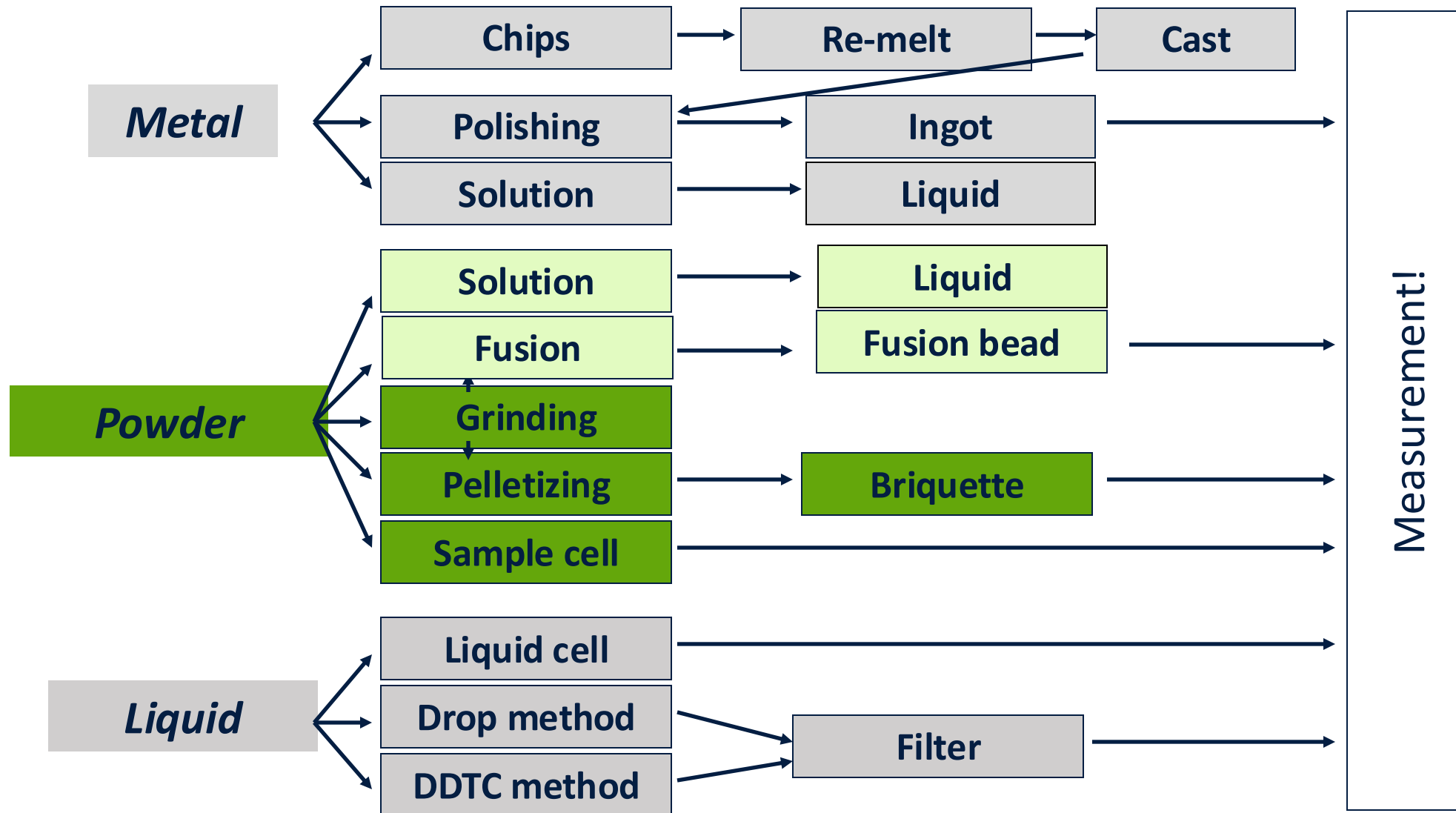
1. Inductively Coupled Plasma Mass Spectrometry (ICP-MS)
2. X-ray Photoelectron Spectroscopy (XPS)
3. X-ray Fluorescence (XRF)
4. Others!

# Sample Preparation

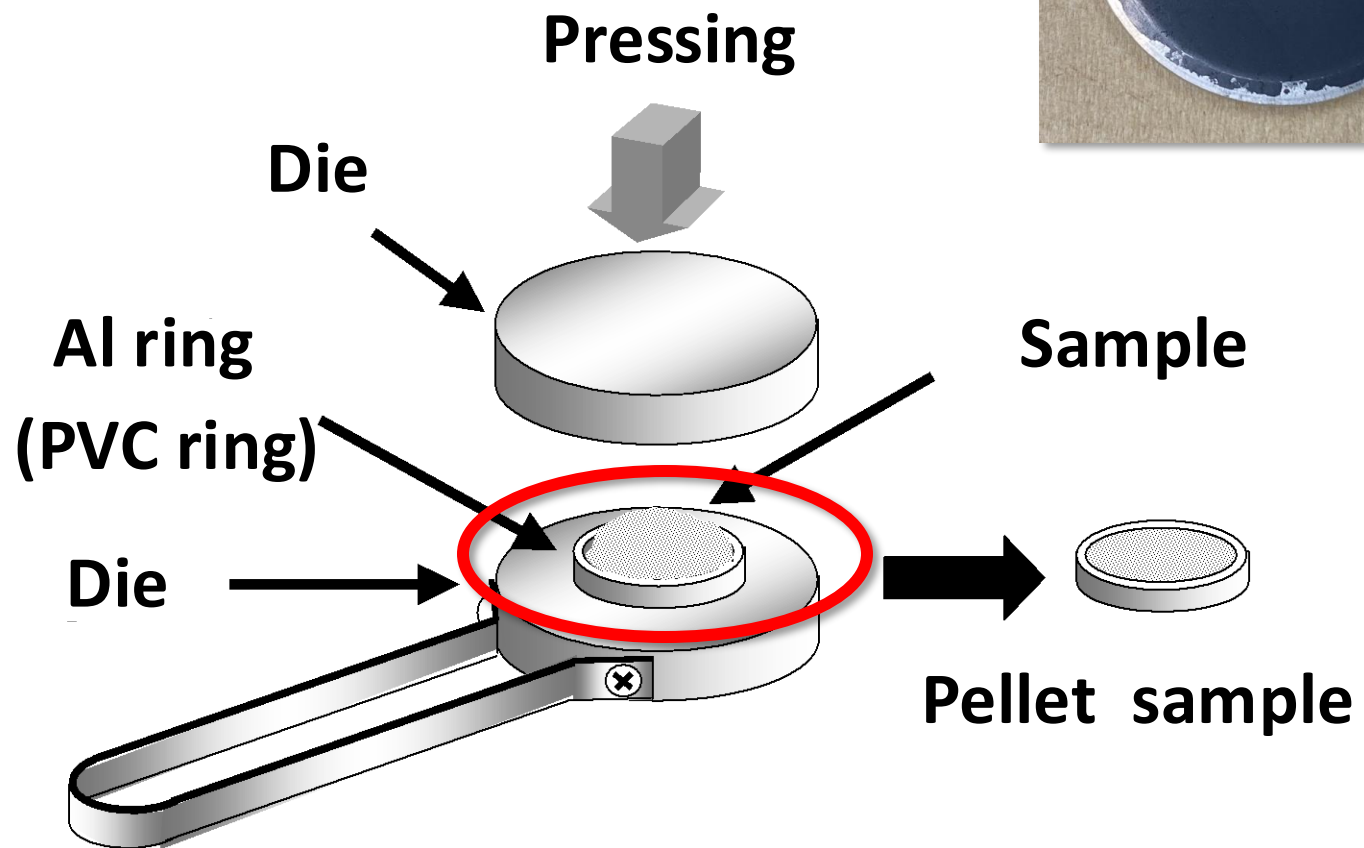
1. Solids
2. Powders
3. Liquids



# Sample Preparation: Overview



# Sample Preparation: Pressed Pellet





# Wide Range of Analytes

1 1a																	18 0
1 H 1.008	2 IIa											13 IIIb	14 IVb	15 Vb	16 VIb	17 VIIb	18 VIIIb
3 Li 6.94	4 Be 9.0122 Ka RX85 0.111											5 B 10.81 67.6 RX85 0.19	6 C 12.011 31.6 RX61 0.252	7 N 14.007 31.6 RX45 0.4	8 O 15.999 23.66 RX35 0.51	9 F 18.998 18.32 RX25 0.664	10 Ne 20.180
11 Na 22.990 11.91 RX25 1.07	12 Mg 24.305 9.889 RX25 1.30	3 IIIa	4 IVa	5 Va	6 VIa	7 VIIa	8 VIIIa	9 VIIIa	10 VIIIa	11 Ib	12 IIb	13 Al 26.982 8.336 144.61° 1.55 PET	14 Si 28.085 7.216 109.64° 1.83 PET	15 P 30.974 8.155 141.16° 2.14 Ge	16 S 32.06 8.155 110.82° 2.47 Ge	17 Cl 35.45 4.729 92.67° 2.82 Ge	18 Ar 39.948
19 K 39.098 3.742 LIF(200) 136.89° 3.59	20 Ca 40.078(4) 3.359 LIF(200) 113.09° 4.00	21 Sc 44.956 3.032 LIF(200) 97.70° 4.49	22 Ti 47.867 2.750 LIF(200) 86.11° 4.95	23 V 50.942 2.505 LIF(200) 76.91° 5.45	24 Cr 51.996 2.291 LIF(200) 69.33° 5.98	25 Mn 54.938 2.103 LIF(200) 62.95° 6.54	26 Fe 55.845(2) 1.937 LIF(200) 57.50° 7.10	27 Co 58.933 1.342 LIF(200) 52.77° 7.71	28 Ni 58.693 1.659 LIF(200) 48.65° 8.29	29 Cu 63.546(3) 1.436 LIF(200) 45.01° 8.86	30 Zn 65.38(2) 1.436 LIF(200) 41.78° 9.65	31 Ga 69.723 1.341 LIF(200) 38.90° 19.4	32 Ge 72.630(3) 1.255 LIF(200) 36.31° 11.1	33 As 74.922 1.177 LIF(200) 33.98° 11.9	34 Se 78.971(8) 1.106 LIF(200) 31.87° 12.7	35 Br 79.904 1.041 LIF(200) 29.95° 13.5	36 Kr 83.798(2)
37 Rb 85.468 0.9269 LIF(200) 26.80° 15.7	38 Sr 87.62 0.8766 LIF(200) 25.13° 16.1	39 Y 88.906 0.8302 LIF(200) 23.78° 17.9	40 Zr 91.224(2) 0.7873 LIF(200) 22.54° 18.0	41 Nb 92.906 0.7476 LIF(200) 21.39° 19.0	42 Mo 95.95 0.7107 LIF(200) 20.32° 20.0	43 Tc 99 0.674 LIF(200) 19.33° 21.1	44 Ru 101.07(2) 0.6445 LIF(200) 18.41° 22.1	45 Rh 102.91 0.6147 LIF(200) 17.55° 23.2	46 Pd 106.42 0.5869 LIF(200) 16.75° 24.4	47 Ag 107.87 0.5608 LIF(200) 16.00° 25.5	48 Cd 112.41 0.5365 LIF(200) 15.30° 26.7	49 In 114.82 0.5136 LIF(200) 14.64° 27.9	50 Sn 118.71 0.4820 LIF(200) 14.03° 29.1	51 Sb 121.76 0.4718 LIF(200) 13.45° 30.4	52 Te 127.60(3) 0.4328 LIF(200) 12.90° 31.8	53 I 126.90 0.4348 LIF(200) 12.39° 33.2	54 Xe 131.29
55 Cs 132.91 0.4018 LIF(200) 11.44° 35.9	56 Ba 137.33 2.776 LIF(200) 87.13° 5.99	57-71 Lanthanoids	72 Hf 178.49(2) 1.569 LIF(200) 45.86° 11.3	73 Ta 180.95 1.522 LIF(200) 44.40° 11.7	74 W 183.84 1.476 LIF(200) 43.00° 12.1	75 Re 186.21 1.433 LIF(200) 41.67° 12.5	76 Os 190.23(3) 1.391 LIF(200) 40.41° 13.0	77 Ir 192.22 1.352 LIF(200) 39.20° 13.4	78 Pt 195.08 1.313 LIF(200) 38.04° 13.9	79 Au 196.97 1.277 LIF(200) 36.94° 14.4	80 Hg 200.59 1.242 LIF(200) 35.89° 14.8	81 Tl 204.38 1.207 LIF(200) 34.88° 15.3	82 Pb 207.2 1.175 LIF(200) 33.92° 15.8	83 Bi 208.98 1.144 LIF(200) 32.99° 16.4	84 Po 210 1.114 LIF(200) 32.10° 16.9	85 At 210 1.085 LIF(200) 31.25° 17.5	86 Rn 222
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57-71 Lanthanoids			57 La 138.91 2.665 LIF(200) 82.88° 6.26	58 Ce 140.12 2.561 LIF(200) 78.98° 6.54	59 Pr 140.91 2.463 LIF(200) 75.40° 6.83	60 Nd 144.24 2.370 LIF(200) 72.10° 7.12	61 Pm 145 2.283 LIF(200) 69.03° 7.45	62 Sm 150.36(2) 2.199 LIF(200) 66.20° 7.73	63 Eu 151.96 2.120 LIF(200) 63.54° 8.04	64 Gd 157.25(3) 2.046 LIF(200) 61.08° 8.37	65 Tb 158.93 1.976 LIF(200) 58.77° 8.70	66 Dy 162.50 1.909 LIF(200) 56.57° 9.03	67 Ho 164.93 1.845 LIF(200) 54.52° 9.36	68 Er 167.26 1.785 LIF(200) 52.58° 9.73	69 Tm 168.93 1.726 LIF(200) 50.77° 10.1	70 Yb 173.05 1.672 LIF(200) 49.04° 10.5	71 Lu 174.97 1.619 LIF(200) 47.41° 10.9
89-103 Actinoids			89 Ac 227 0.980 LIF(200) 28.15° 19.8	90 Th 232.04 0.956 LIF(200) 27.45° 20.5	91 Pa 231.04 0.933 LIF(200) 26.78° 21.1	92 U 238.03 0.911 LIF(200) 26.13° 21.7	93 Np 237	94 Pu 239	95 Am 243	96 Cm 247	97 Bk 247	98 Cf 252	99 Es 252	100 Fm 257	101 Md 258	102 No 259	103 Lr 262

- Alkali metals
- Alkaline - earth metals
- Transition metals
- Rare - earth elements
- Actinoid elements
- Other metals
- Other nonmetals
- Halogens
- Noble gases

Atomic Number → 45 Rh ← Symbol

102.91 ← Atomic Weight

0.6147 ← Spectrum Wavelength(Å)

17.55° ← Analyzing Crystal 2θ Angle

23.2 ← Excitation Voltage(kV)

# Ease of Use

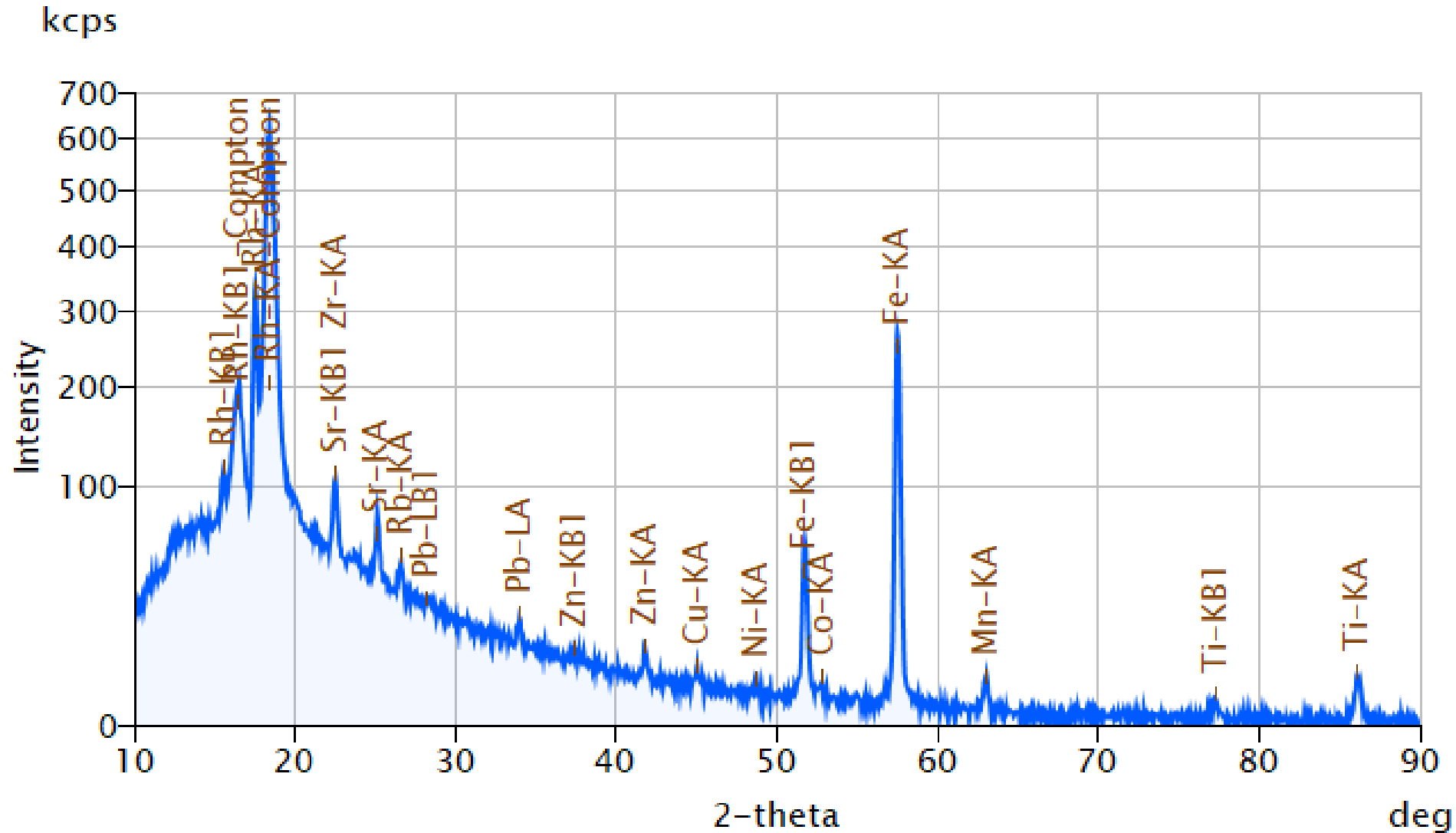
Technique	ICP-MS	XRF
Prep time	Hours +	5-15 minutes
Prep materials needed	<b>Acids</b> , glassware, centrifuge, digesting block, hot plate, etc.	Press, die assembly
Analysis time	Minutes	<1-30 minutes
Sample recovery	Typically no	Yes
Calibration setup	Individual element	All elements
Calibration time	Hours +	Hour
Calibration upkeep	Daily +	Every 6 months
<b>Operator</b>	<b>Chemist</b>	<b>Anyone</b>



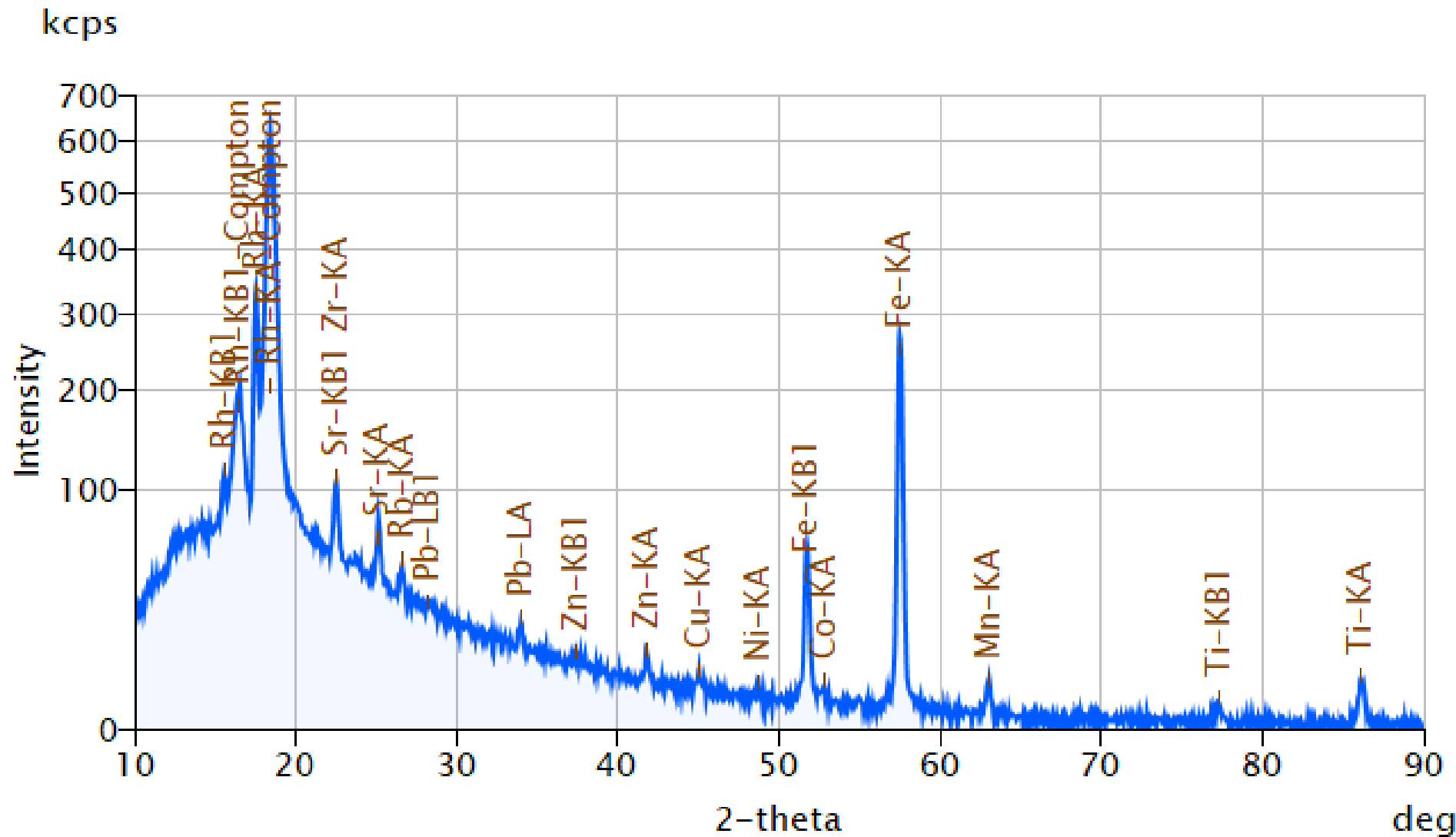
# Types of Analyses

1. Qualitative (Scanning for all analytes!)
2. Semi-quantitative
3. Quantitative

# Types of Analyses: Qualitative



# Types of Analyses: Semi-quantitative



Comp.	Result (mass%)
Na2O	0.66
MgO	1.33
Al2O3	19.93
SiO2	42.09
P2O5	0.83
SO3	2.01
Cl	0.17
K2O	4.18
CaO	8.58
TiO2	2.91
MnO	0.25
Fe2O3	16.24
NiO	0.05
CuO	0.05
ZnO	0.09
Rb2O	0.07
SrO	0.18
ZrO2	0.13
PbO	0.20
SnO2	0.07

# Types of Analyses: Quantitative



Canada Natural Resources Canada / Ressources naturelles Canada

CCRMP Canadian Certified Reference Materials Project

PCMRPC Projet canadien de matériaux de référence certifiés

## Certificate of Analysis

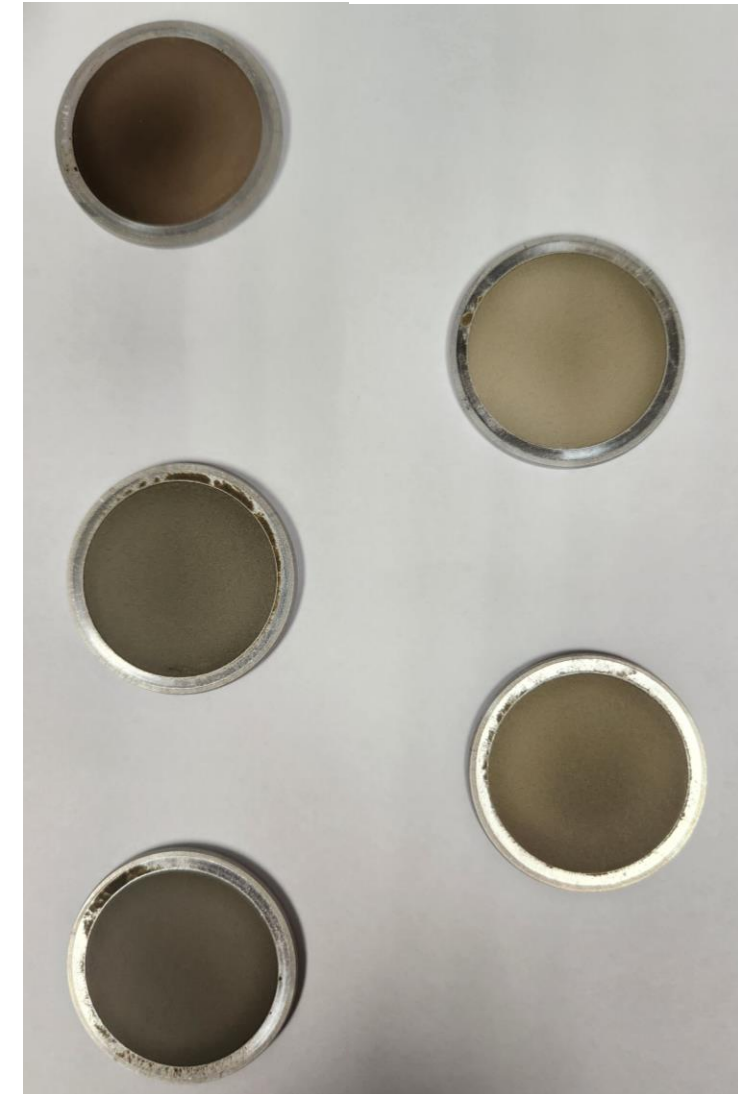
First issued: April 2015 Version: April 2015

### RTS-5

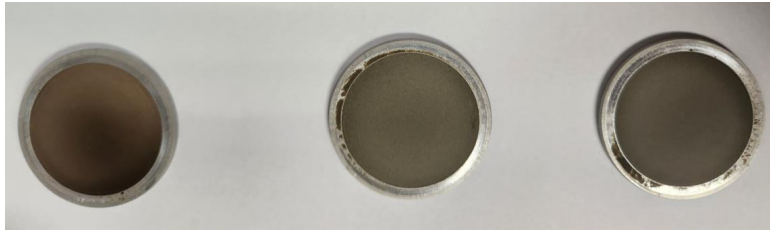
Certified Reference Material for a Nickel-Copper-Gold Tailings

**Table 1 – RTS-5 Certified Values**  
*note: For some elements, some methods do not produce a complete recovery. Where possible, certified, provisional or informational values are provided for those cases.*

Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
Ag	µg/g	1.50	0.07	0.10	0.04
Al (FUS) <sup>a</sup>	%	6.25	0.04	0.13	0.08
As	µg/g	1286	27	84	28
Au (no AD2) <sup>b</sup>	µg/g	0.408	0.017	0.017	0.007



# Types of Analyses: Quantitative



Natural Resources Canada / Ressources naturelles Canada

CCRMP / Canadian Certified Reference Materials Project

PCMR / Projet canadien de matériaux de référence certifiés

## Certificate of Analysis

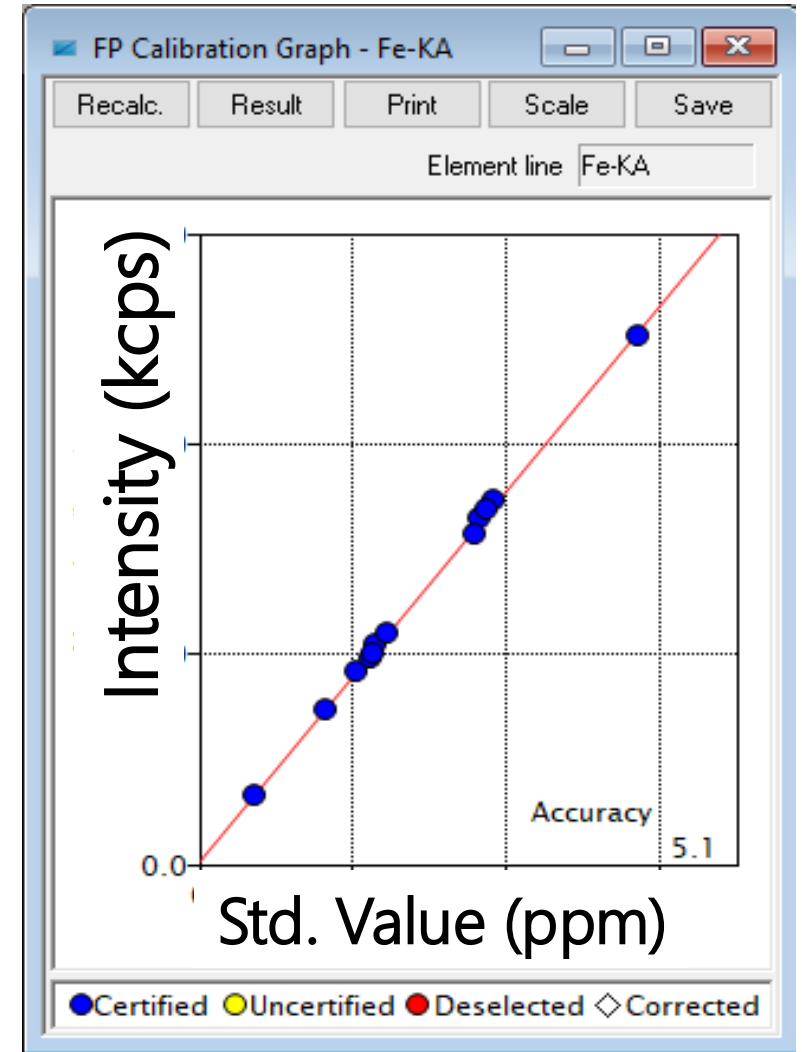
First issued: April 2015      Version: April 2015

### RTS-5

Certified Reference Material for a Nickel-Copper-Gold Tailings

**Table 1 – RTS-5 Certified Values**  
note: For some elements, some methods do not produce a complete recovery. Where possible, certified, provisional or informational values are provided for those cases.

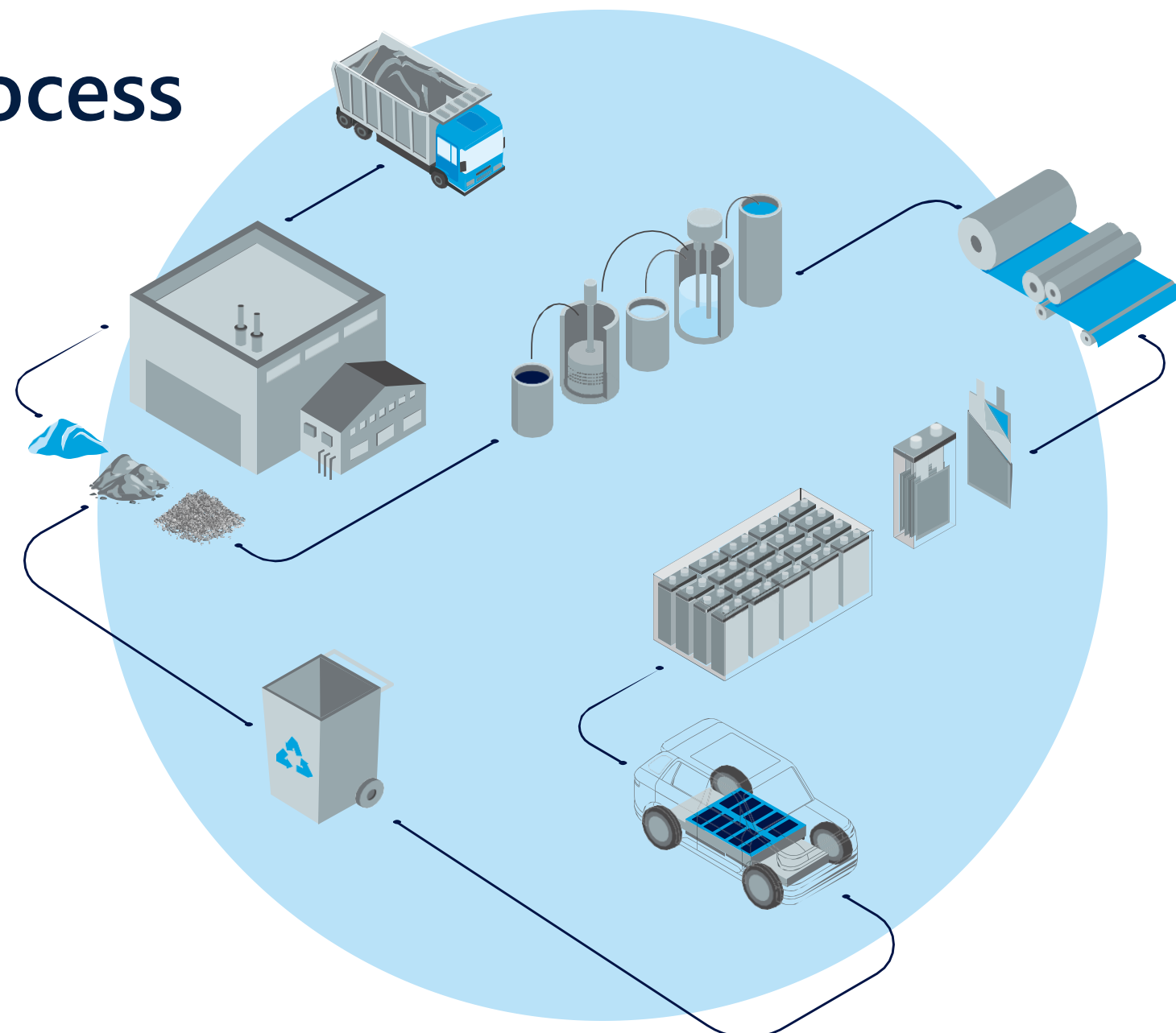
Element	Units	Mean	Within-lab Standard Deviation	Between-labs Standard Deviation	95% Confidence Interval of Mean
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As	µg/g	1286	27	84	28
Au (no AD2) <sup>b</sup>	µg/g	0.408	0.017	0.017	0.007



# Where is XRF used?

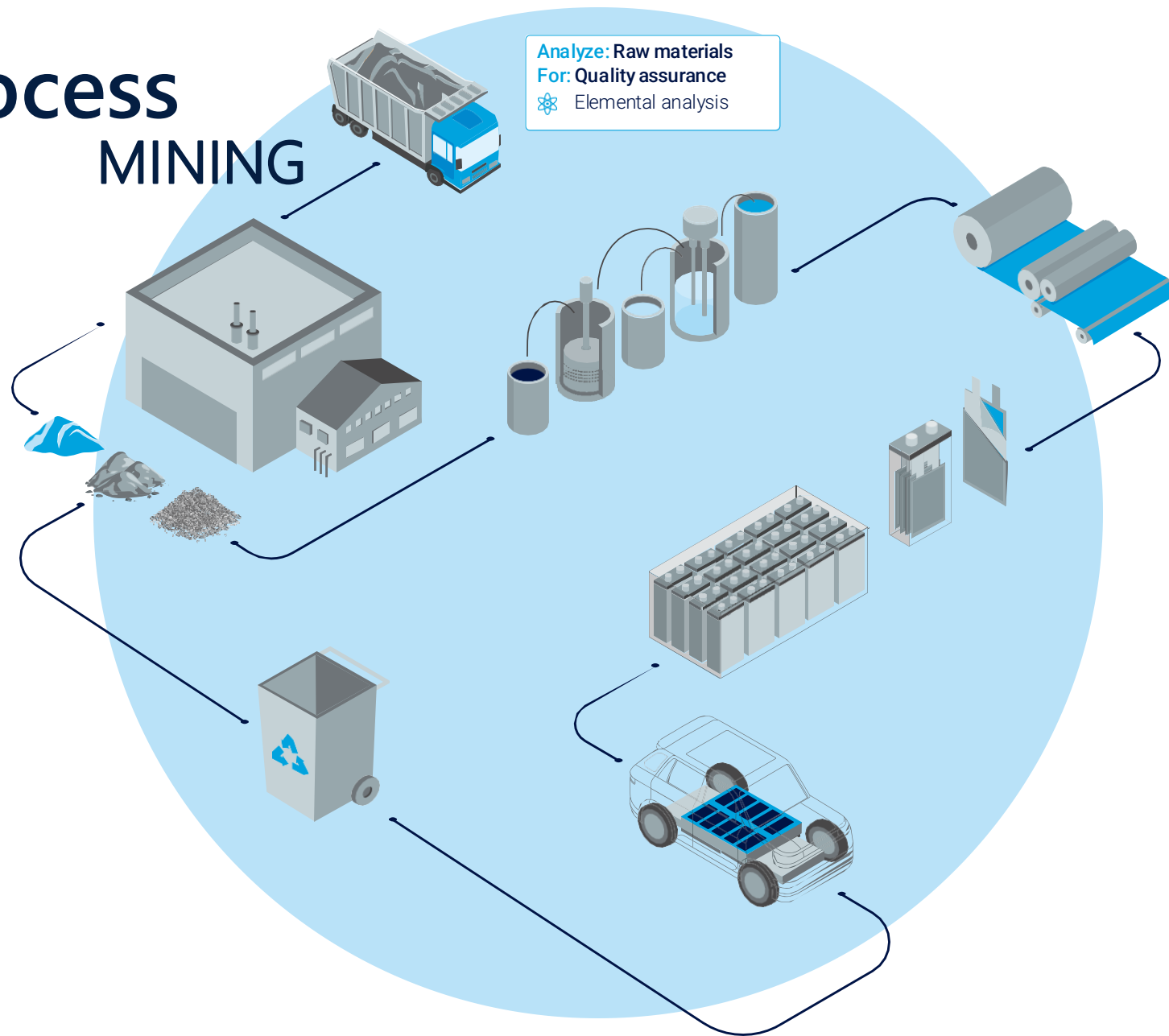


# The Process



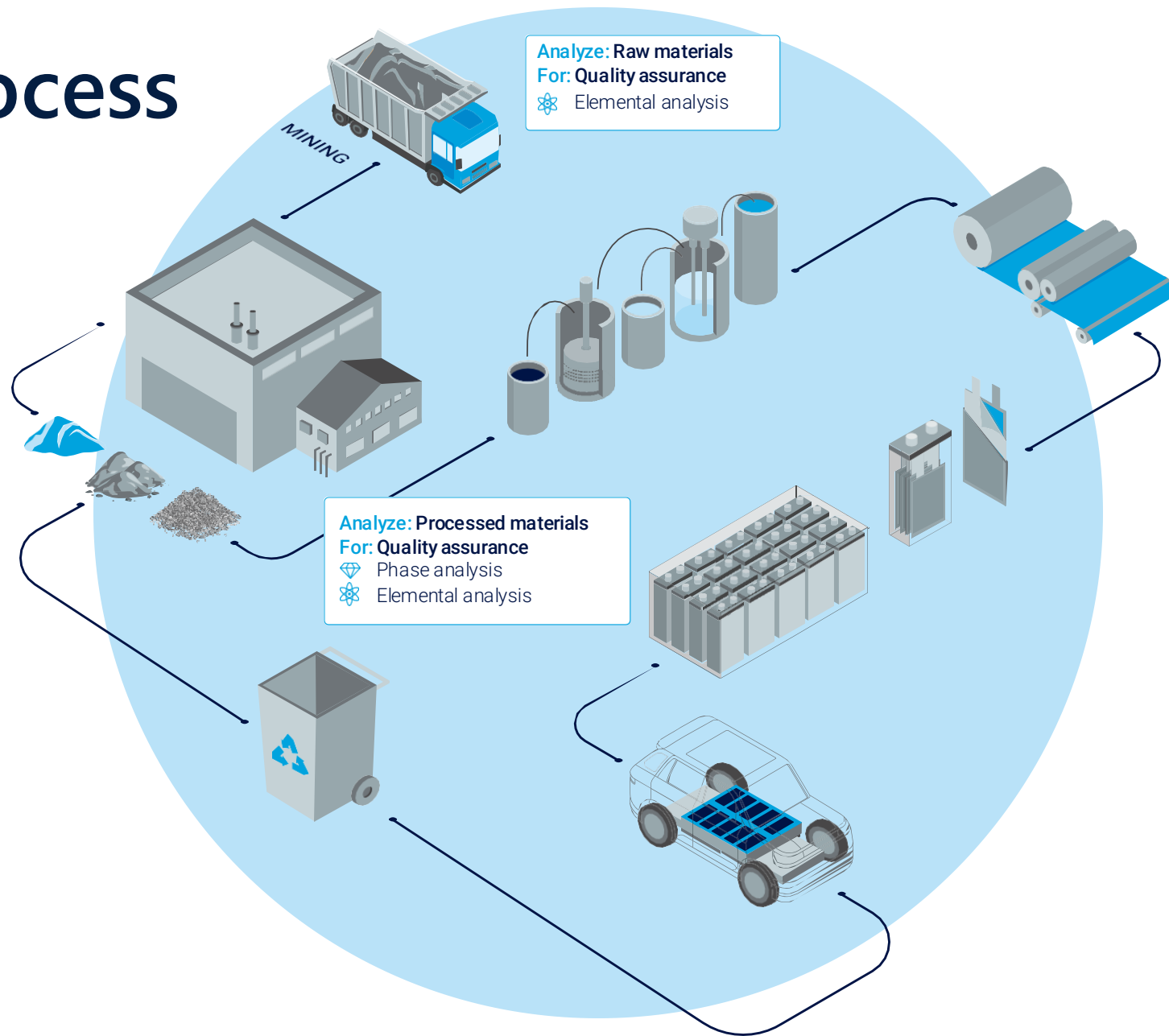
# The Process

## MINING

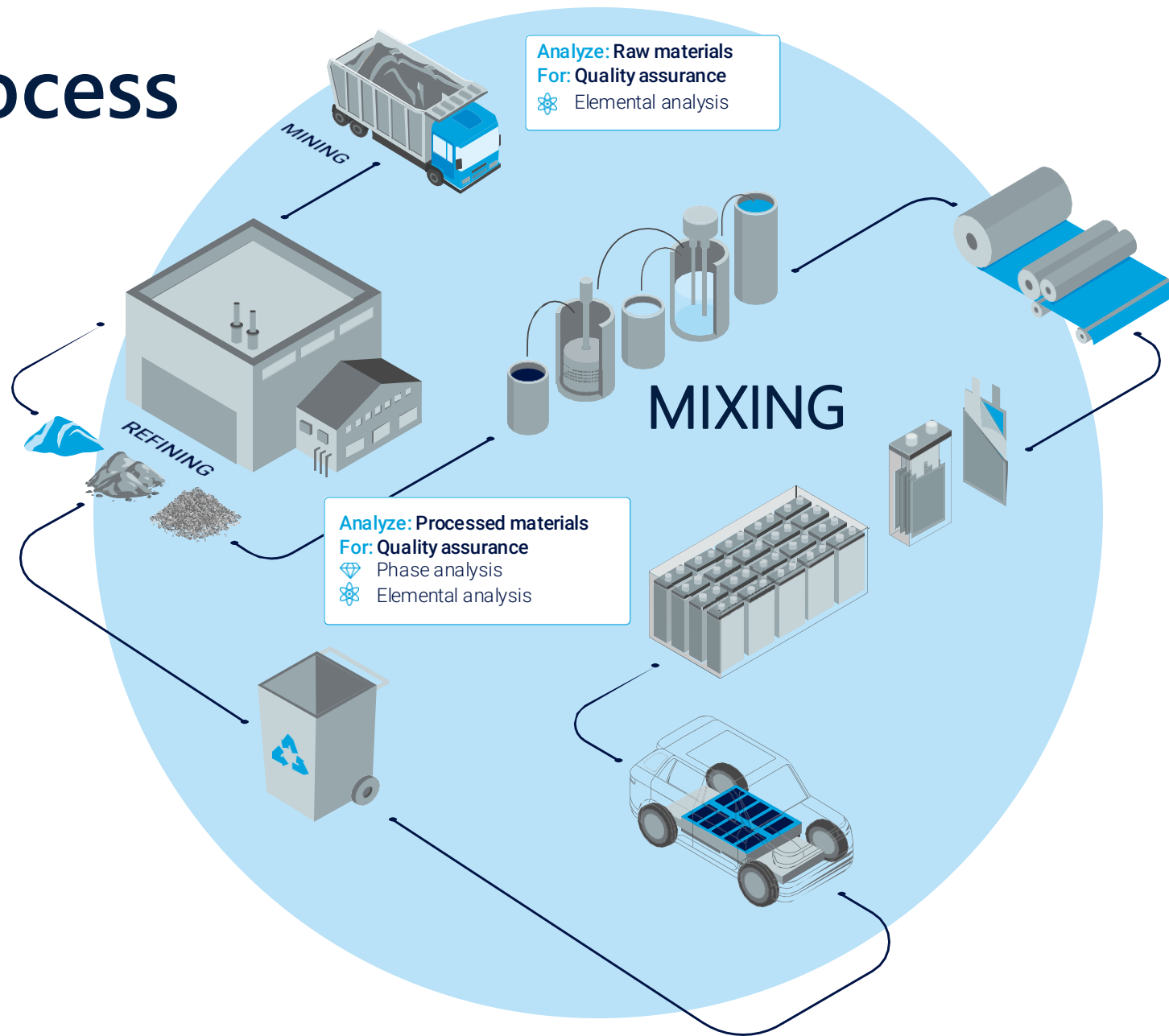


# The Process

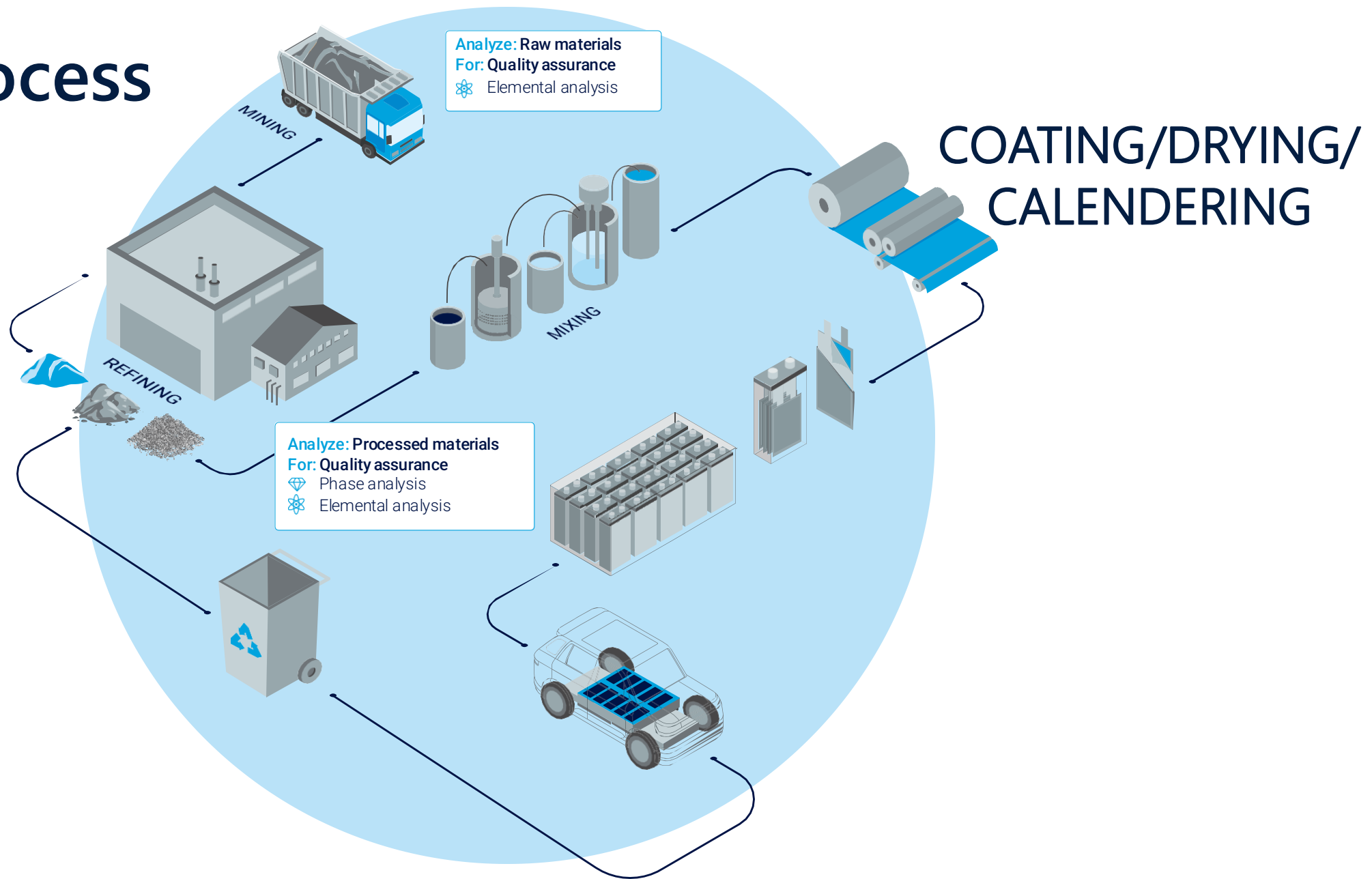
REFINING



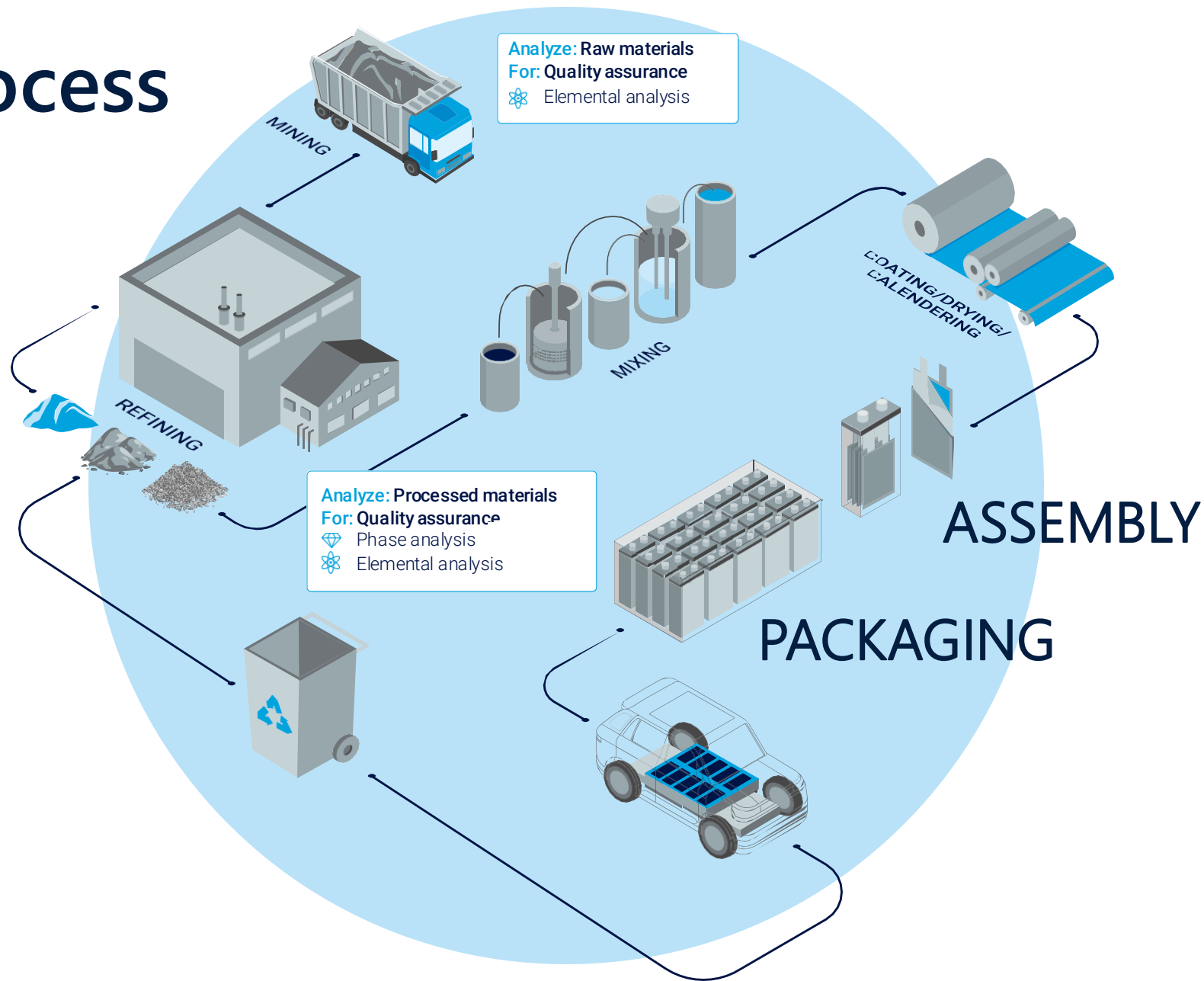
# The Process



# The Process

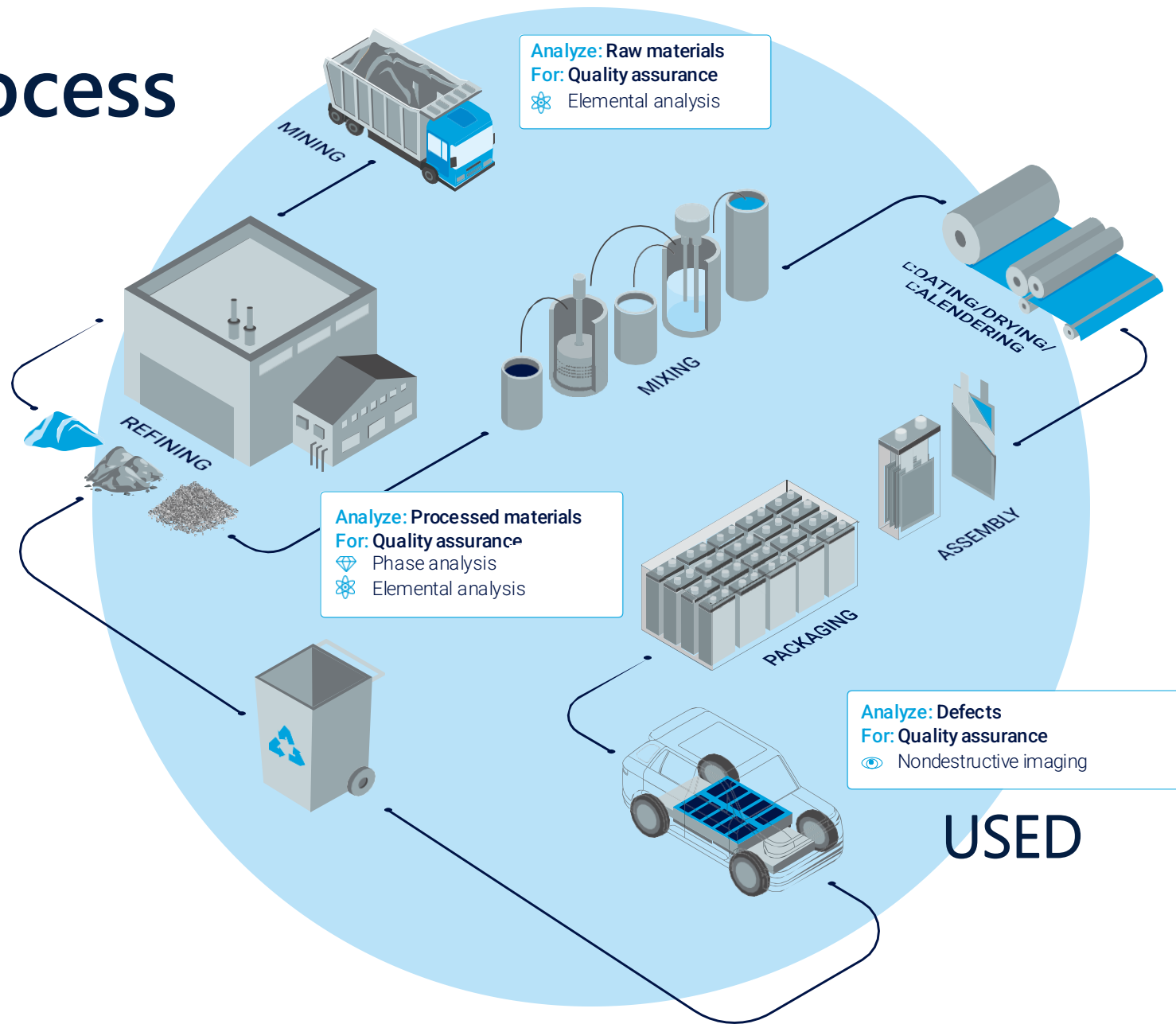


# The Process

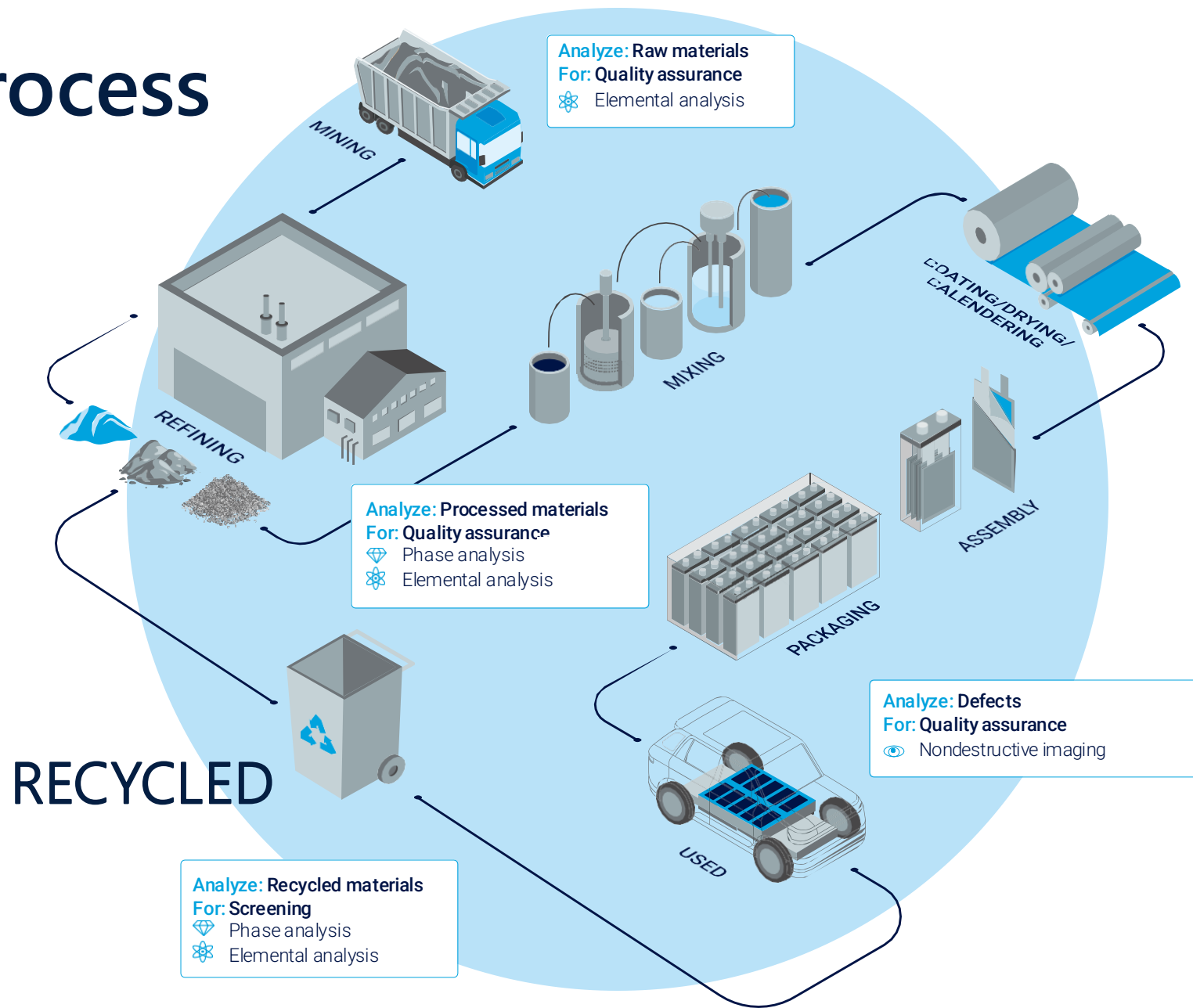




# The Process



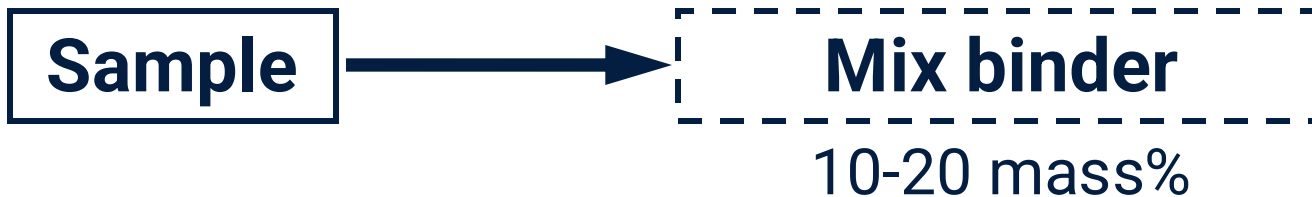
# The Process



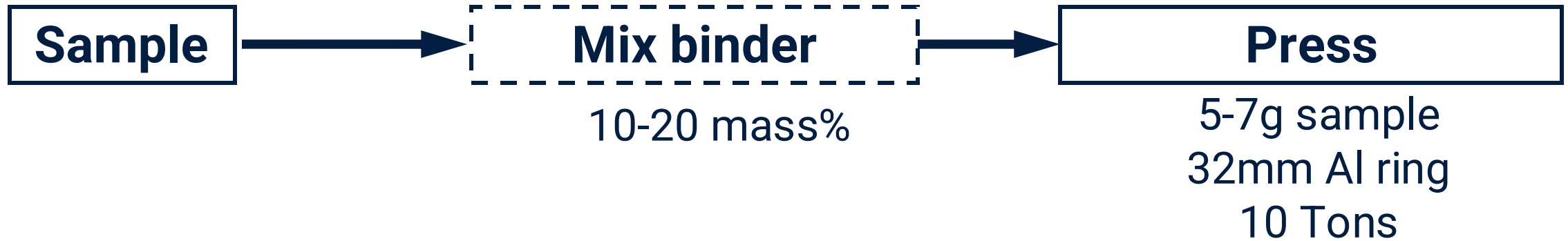
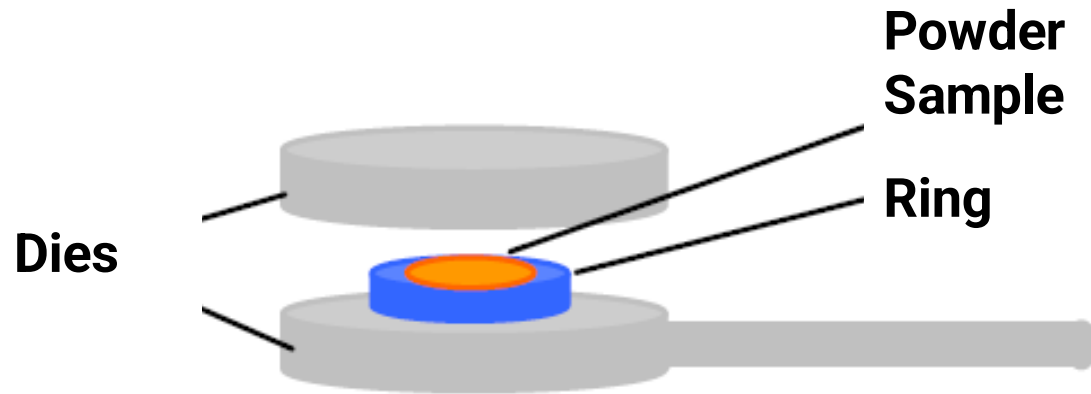
# How is XRF used?

# NCM Cathode Example

# NCM Cathode Example: Prep



# NCM Cathode Example: Prep



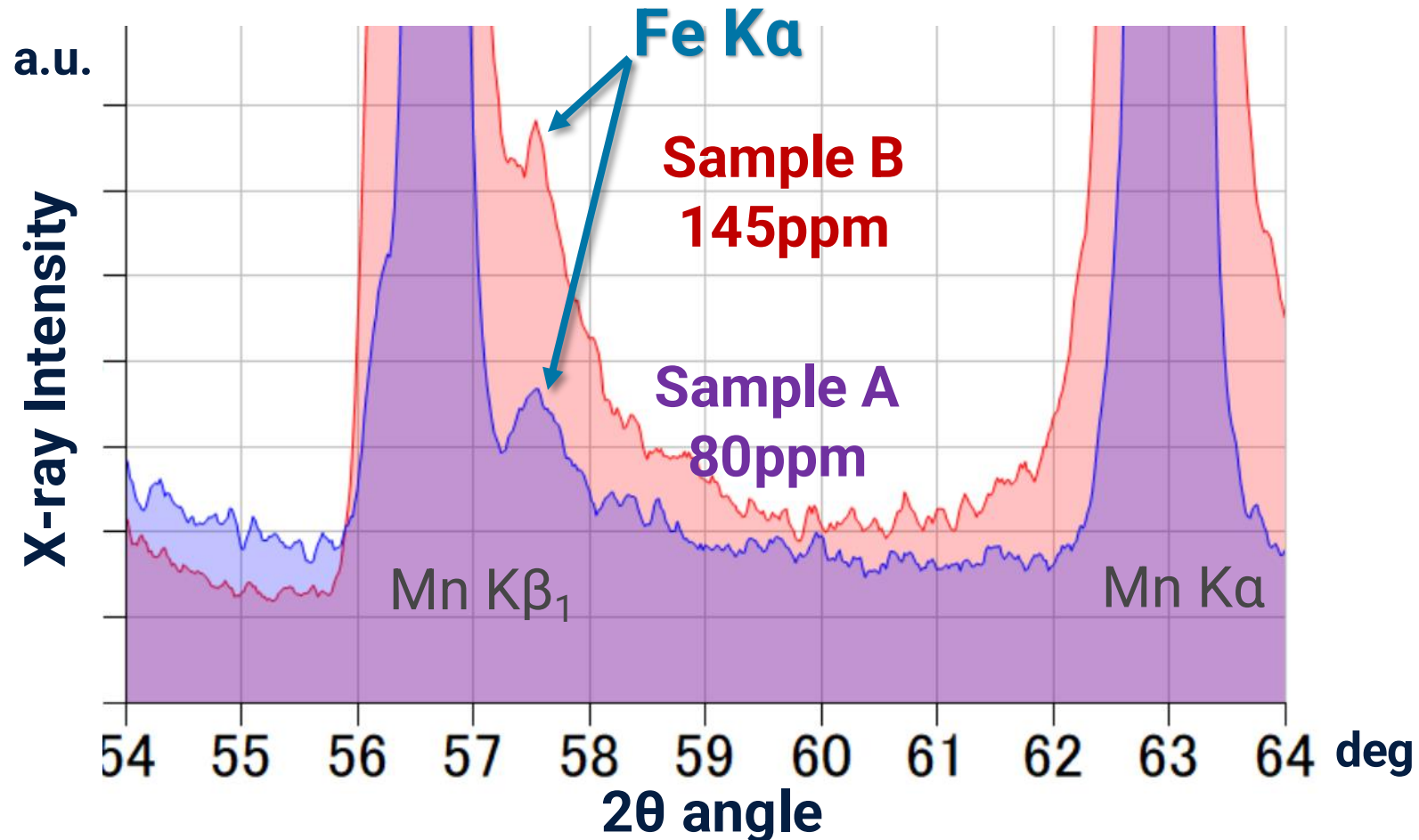
# NCM Cathode Example: Semi-Quantitative Results

		Ni	Co	Mn
Sample A	WDXRF	0.846	0.098	0.056
	ICP	0.85	0.10	0.05
Sample B	WDXRF	0.490	0.201	0.309
	ICP	0.50	0.20	0.30

Unit : mol

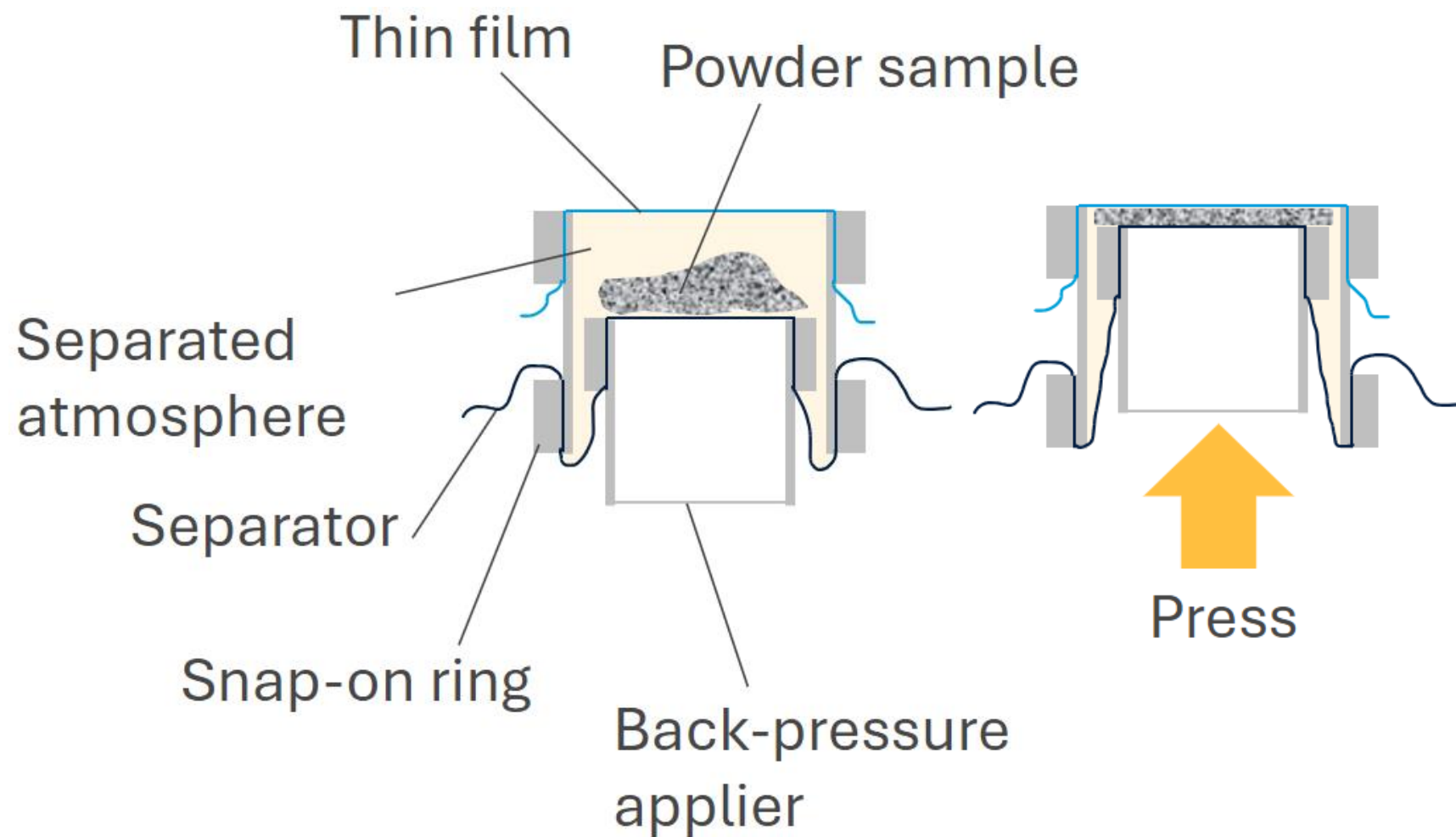


# NCM Cathode Example: Impurity Analysis

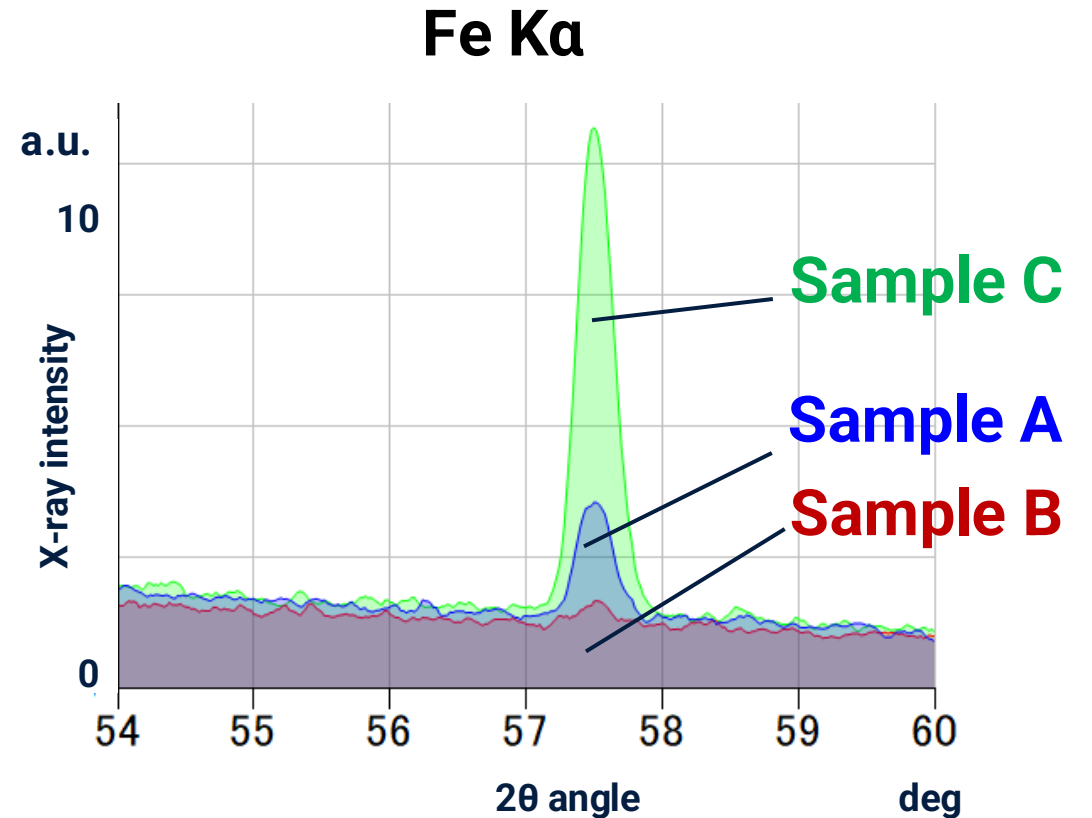
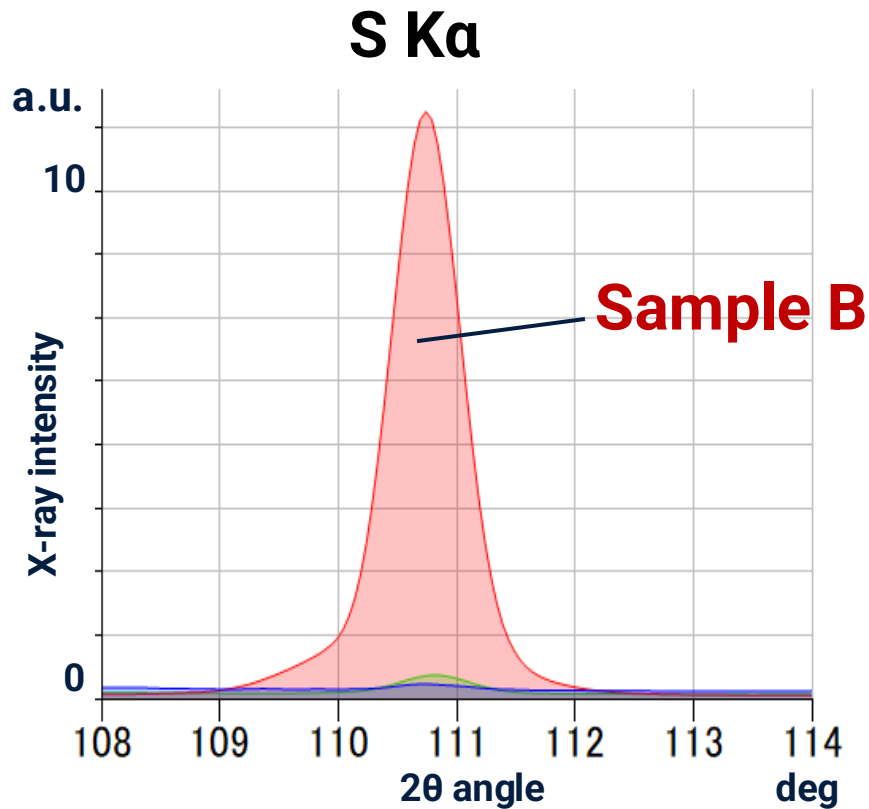


# Graphite Anode Example

# Graphite Anode Example: Prep



# Graphite Anode Example: Semi-quantitative Results



# Graphite Anode Example: Semi-quantitative Results

	Na	Mg	Al	S	Ca	Fe	Zr
Sample A	83	249	28	38	73	39	13
Sample B	N.D.	975	24	2958	41	6	N.D.
Sample C	81	242	42	76	81	180	4

Unit : ppm

# Black Mass Recycled Material Example

# Black Mass Recycled Screening Example: Setup



NEX QC+  
QuantEZ EDXRF



# Black Mass Recycled Screening Example: Semi-quantitative Results

Element	Result (mass%)	Stat. error
Co	13.556	0.008
Mn	9.963	0.008
Ni	16.163	0.008
Al	6.156	0.009
Cu	6.605	0.009
Fe	2.104	0.003
Si	0.7577	0.0020
P	2.099	0.002
S	0.1275	0.0004
Cl	0.0317	0.0002
K	0.2074	0.0067
Ca	0.4066	0.0050

Ti	0.4774	0.0030
V	0.0527	0.0012
Cr	0.0382	0.0008
Zn	0.3678	0.0013
As	0.0028	0.0002
Zr	0.1358	0.0003
Ag	0.0026	0.0001
Cd	0.1214	0.0004
Sn	0.0766	0.0003
Sb	0.0256	0.0003
Ba	0.0248	0.0006
Pb	0.0528	0.0005

# Polling Question #3



Microsoft Stock

# Summary

## 1. What is XRF?

- Elemental analysis covering a wide range
- Variety of instruments
- Variety of analysis “flavors”

# Summary

## 2. Why use XRF?

- Non-destructive
- Ease of prep and use
- Variety of preparations to meet any needs
- Speed of analysis

# Summary

## 3. Where is XRF used?

- Mining
- Refining
- Recycling

# Summary

## 4. How is XRF used?

- Black mass recycling screening
- Graphite anode
- NCM cathode powder
- Many more!!!

# Questions and Answers





We'll follow up with  
your questions.



Recording will be  
available tomorrow.



[Check out our new  
Ebook!](#)

# Thank you!

